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ELASTIC CONSTANT MEASUREMENTS OF POLYCRYSTALLINE
INFRARED WINDOW MATERIALS

S. A. Kulin, et al

Air Force Cambridge Research Laboratories
Hanscom Air Force Base, Massachusetts

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The experimental results obtained were, in some cases, compared with various calculation approximations which yield polycrystalline elasticity parameters from single crystal data.

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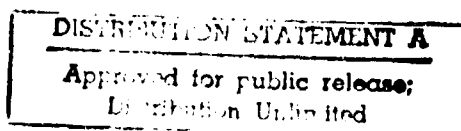
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ELASTIC CONSTANT MEASUREMENTS OF
POLYCRYSTALLINE INFRARED WINDOW MATERIALSS. A. Kulin
ManLabs, Inc.R. Salzbrenner
Massachusetts Institute of TechnologyH. Posen
Air Force Cambridge Research LaboratoriesAbstract

Although the critical property of laser windows is the optical absorption, other parameters are necessary to the selection of the appropriate window material. Theoretical criteria developed by Horrigan and Bendow require a knowledge of the elastic properties of the material. In particular, Young's Modulus and Poisson's ratio are an integral part of these criteria and are also a prerequisite for any engineering design. Unfortunately, in many cases, the elastic constants of the materials are not available and when available are either for single crystals or are of doubtful validity.

In this work elastic constants of some infrared window materials were calculated from ultrasonic measurements using a pulse overlap technique. Velocity measurements were determined in various directions in CVD zinc selenide and in hot forged KCl alloy windows. These measurements permitted the determination of Young's Modulus, the shear modulus, the bulk modulus, and Poisson's ratio. Some anisotropy was found in the elastic properties of these fabricated or "as-grown" infrared windows. For example, in the case of the chemically vapor deposited ZnSe which exhibits a small amount of elastic anisotropy, the elastic properties were found to be isotropic in all directions normal to the growth direction. The modulus values observed for directions in the plane of the zinc selenide window were found to change by approximately two percent when measured in directions parallel to the plate growth direction.

The experimental results obtained were, in some cases, compared with various calculation approximations which yield polycrystalline elasticity parameters from single crystal data.



INTRODUCTION

The appreciation that materials to be used in high energy laser optics must represent a compromise of a variety of materials properties was recognized early in the development of window materials appropriate for the 10.6 μ m lasers. Theoretical criteria developed for window materials selection by Sparks (1), Horrigan (2) and Bendow (3) include not only the absorption at the relevant wavelength but also elasto-optic, mechanical strength terms and the thermal properties of the materials. These theoretical criteria are usually embodied in figures of merit. The variety of figures of merit reflect models based on either different failure modes and/or different operating conditions. Table 1, from Horrigan (2), illustrates the variety of such figures of merit assuming bulk absorption only. In those instances where some optical distortion can be tolerated, fracture becomes the limiting factor, and it is desirable to select materials with a high critical failure stress, σ_c . Although σ_c is not explicitly included in those figures of merit which regard optical distortion as the limiting factor, the yield strength is also an important parameter in that distortions beyond the elastic limit induce permanent optical distortion.

Thus the emphasis over the past few years has been directed towards improving σ_c , the critical failure stress and yield strengths by a variety of techniques such as pressure induced recrystallization (4,5), alloying (6), casting (7), etc. in which polycrystalline aggregates are developed to provide the desired strength and dimensions. Little emphasis has been given to considerations of the elasto-mechanical properties such as Young's Modulus, E, and Poisson's ratio, σ . These parameters are explicitly involved in all figures of merit criteria, regardless of whether they are based on fracture criteria or optical distortion criteria. Perhaps their most important role in materials selection or design consideration is that they affect these figures of merit inversely to those of the strength parameters such as σ_c , the critical failure stress and yield strengths.

Window materials of the cubic crystalline class do not show anisotropy in thermal expansion coefficients or refractive index, which can be described in terms of second rank tensors (8), but other properties involving the moduli of elasticity require fourth rank tensors for their complete description. Thus for cubic crystals the expression for Young's modulus, E,

$$1/E = S_{11} - 2[S_{11}S_{12} - 0.5S_{44}] \times (\ell_1^2\ell_2^2 + \ell_1^2\ell_3^2 + \ell_2^2\ell_3^2)$$

where S_{ij} are the elastic compliances and the ℓ_i are the direction cosines of the stress axis with the cubic crystallographic

axes, illustrates that even for single crystal cubic materials there are variations in the modulus with crystalline direction. Hence, the maximum modulus value is in the $\langle 111 \rangle$ direction and the minimum value is in the $\langle 100 \rangle$ direction. Any property which exhibits anisotropy in a single crystal will consequently be anisotropic in a non-randomly oriented polycrystalline aggregate. Polycrystalline windows are known to exhibit texture by virtue of the processing procedures (9) and thus should show (elastic) anisotropy.

In this paper we report on elastic moduli measurements in some polycrystalline laser window materials. For the polycrystalline samples studied one can correlate the elastic moduli with the texture properties resulting from the fabrication methods.

It is also shown that the as-measured properties of polycrystalline aggregates may differ significantly from those calculated by any of the accepted averaging methods utilizing single crystal parameters.

2. EXPERIMENTAL METHODS

The velocity of 5MHz waves (shear and longitudinal) traveling through the specimen were measured by the pulse-echo-overlap method. This method (10,11) is a highly accurate process for measuring the velocity of ultrasonic waves in materials. The absolute accuracy arises from the fact that the method is capable of precisely measuring the time from any cycle of one echo to the corresponding cycle of the next echo. In the present work the pulse overlap method was operated with broad band pulses which insure that the proper overlap can be unambiguously obtained within the broad band.

A block diagram of the equipment for the pulse-echo measurements with broad band pulses is shown in Figure 1. The principle of the measurement is to overlap the two signals of interest on the oscilloscope by driving the x-axis with a frequency whose period is the travel time between these two signals. Each of the two signals appears on alternating sweeps of the oscilloscope. The x-axis sweep frequency is supplied by a continuous wave (CW) oscillator. Overlap of the two echoes on the oscilloscope screen is achieved by adjusting the CW frequency which is then counted by a frequency counter and the travel time between the pulses is obtained as the reciprocal of this value. From a knowledge of transit times and specimen dimensions, velocities can then be calculated.

The samples used in this study were parallelepipeds cut

from "as-fabricated"^{*} or "as-grown" specimens of various window materials supplied by the Air Force Weapons Laboratory. All materials were polycrystalline. Six faces of each specimen were polished with each set of opposite faces polished parallel to ± 0.0002 inch. Most specimens were approximately 3/4 inch by 3/4 inch by the supplied thickness which was somewhat reduced by the grinding and polishing operations employed. Pre-packaged wear-face 5MHz transducers were coupled to each face and the velocity of the broad band wave was measured. In order to calculate the various elastic constants in these polycrystalline samples, both longitudinal and shear wave velocities must be measured. From the measured wave velocity values, dynamic values for Young's Modulus, E; the shear modulus, G; the bulk modulus, K; and Poisson's ratio, σ , can be calculated using the following relations (12):

$$E = \rho v_s^2 \left| \frac{3v_l^2 - 4v_s^2}{v_l^2 - v_s^2} \right|$$

$$G = \rho v_s^2$$

$$K = \rho/3(3v_l^2 - 4v_s^2)$$

$$\sigma = \frac{1}{2} \left(\frac{v_l^2 - 2v_s^2}{v_l^2 - v_s^2} \right)$$

where ρ is the density of the material, v_s is the shear wave velocity and v_l is the longitudinal wave velocity. Figure 2 schematically depicts the ultrasonic wave modes employed in these measurements.

The densities of the specimens of the various materials were experimentally determined using standard precision weighing techniques. In this work, the density was calculated from weight measurements of each specimen both in air and in isopropyl alcohol.

Certain small errors are introduced in this experimental measurement of elastic constants. In order to minimize transducer-to-specimen error the relative surface area ratio should be approximately 1/2 or less. This is not always possible depending on the final geometry of the PIR processed or CVD grown window materials. The wear-face transducer employed may

^{*} With the exception of the ZnSe (grown by Raytheon Mfg. Co. using CVD methods), the AFWL materials were processed by PIR or Hot Forging techniques.

introduce a maximum error of about 0.1%. Parallelism of final polished opposite specimen faces was held to ± 0.0002 which is well within the error given above. Generally, the electronic errors are quite negligible. The overall accuracy of the method can be considered to be better than 0.5% and in ideal cases one can expect to achieve at least 0.1% total error. The precision of the method is quite good with reproducible values in much better agreement than the accuracy limits given above.

In order to obtain some measure of anisotropy in the window specimens, two different polarizations of the shear waves were used. In this experimental work the shear wave directions employed were, in each case, parallel to corresponding edges of the parallelepiped. It should be borne in mind that these measurements may not yield maximum anisotropy values depending on the geometry of the specimen with respect to both the original window disc and/or the texture of the specimen microstructure.

3. EXPERIMENTAL RESULTS

3.1 Zinc Selenide

The main purpose of this work was to determine the elastic properties and to measure the degree of anisotropy in the elastic moduli in different directions in the chemically vapor deposited ZnSe. The samples were oriented, prepared and measured as shown in Figure 3a, where the growth direction is in the z direction. A summary of the moduli calculated from the velocity of ultrasonic waves (shear and longitudinal) taken in different directions is shown in Table 2. It should be noted that the sample shows essentially no anisotropy for shear waves propagated parallel to the growth direction (i.e. in the z direction). More specifically, shear waves propagated in the growth direction but polarized at 90° to one another travel at the same velocity. When the velocities of shear waves are measured at right angles to the growth direction there is a difference in shear wave velocities, and thus elastic modulus values, for the shear waves polarized at 90° to each other. The measured anisotropy does however display a symmetry. The modulus values calculated for the waves propagated in the x direction (i.e. perpendicular to the growth direction) with the shear wave polarized in the z direction (parallel to growth direction) are very close to the values calculated for the waves propagated in the y direction with the shear wave polarized in the z direction. Likewise the moduli calculated for shear waves polarized perpendicularly to the growth direction are very nearly equal (longitudinal and shear waves propagated both in the x and y directions in the sample).

The type of anisotropy observed may be considered similar to that found in other CVD materials in which one crystal

axis is preferred in the z direction but the other axes have no rotational symmetry about that direction (13).

The measured value obtained for Young's modulus in the z or growth direction of the CVD zinc selenide is 7.4×10^{10} nt/m² (10.94×10^6 psi).

Various methods have been devised for calculation of aggregate or polycrystalline values from single crystal elastic constant data (14). The measured values of the various moduli can be compared with the calculated values obtained by these methods. Such results are shown in Table 3. It is seen that the measured values are bounded by the extremes of the calculated values. Although such calculations do not give accurate values of the elastic moduli, they can nevertheless be employed to yield reasonable estimates of these quantities when no other information is available.

3.2 Alkali Halides

Other laser window materials including Alqloy (KCl-33 m/o KBr), NaCl and KCl-5 m/o KBr, were surveyed in this series of experiments. Samples of these materials were cut from hot forged (PIR) plates. The accuracy (and precision) of these preliminary tests was not as great as in the ZnSe measurements because of the small sample dimensions.

The assumed deformation direction was parallel with the z axis of the samples (Figure 3b). Table 4 shows a compilation of the calculated modulus values for Alqloy. The sample shows no anisotropy between x and y polarized shear waves propagated parallel to the deformation direction. This is similar to the vapor deposited ZnSe. The modulus values for the directions perpendicular to the pressing direction yield moduli in very close agreement to each other. That is, the apparent anisotropy in this Alqloy sample is very small. No values for a shear wave propagated in the y direction with the shear polarization in the z direction were measured because of sample size limitations. Single crystal values for the elastic constants are not available for Alqloy, and thus a comparison of measured values with calculated values could not be made.

The NaCl sample was also formed by hot forging and the sample orientation is similar to that discussed previously. The modulus values calculated from measured wave velocities in NaCl are shown in Table 5. Again no anisotropy is noted for values calculated from shear waves polarized at right angles to one another and propagated in the deformation direction. However, moduli calculated from shear wave velocities propagated in the x and y directions (i.e. perpendicular to the pressing direction) show anisotropy between waves which are polarized at right an-

gles to each other. Waves propagated in the x direction polarized in the y direction yield moduli close to the moduli calculated from waves directed in the y direction polarized in the x direction. Also, the waves directed in the x direction polarized in the z direction yield calculated moduli close to those from waves travelling the the y direction polarized in the z direction. The hot forged NaCl thus has a symmetry of modulus values which is similar to that of vapor deposited ZnSe; the amount of anisotropy is, however, much more exaggerated in this material.

Table 6 gives calculated bulk elastic polycrystalline properties based on the different averaging methods as in Section 3.1. In this case, the calculated values do not agree with the experimentally obtained data. The latter yield values outside the calculated bounds.

An alloy of KCl-5 m/o KBr was also tested with the results given in Table 7. However, a great deal of experimental difficulty was experienced, primarily because of small sample size. Consequently, accuracy and precision were limited to about 5% in this case. The orientation of the sample is again similar to that previously employed.

Only one shear wave polarization could be coupled into each sample face and thus no isotropy data for shear propagation within a given direction could be determined. Within the experimental accuracy no anisotropy between the different directions can be assumed.

Again, since these are alloy data, and no single crystal data is available for either the Alloy or the KCl-5 m/o KBr alloys, comparison cannot be made with calculated polycrystalline data.

4. SUMMARY AND CONCLUSIONS

To summarize the data in order to compare the elastic properties of the various window materials, average modulus values in the deformation direction (or the growth direction in the ZnSe case) are collected in Table 8. As pointed out in the introduction, the figures of merit for window materials always contain the modulus in the denominator. Thus, lowering the modulus produces a higher value for the figure of merit (assuming constancy of other parameters). Table 8 shows the considerable variation in elastic properties found in the infrared window materials.

Values of Young's Modulus, shear modulus, bulk modulus, and Poisson's ratio were determined for a number of infrared window materials using an ultrasonic pulsed-echo-overlap technique and are given in Tables 2-8. The results of these ex-

periments may be summarized as follows:

(1) Some anisotropy in the elastic properties of CVD grown ZnSe was found. The anisotropy of the moduli may be considered similar to that reported for pyrolytic graphite, a material grown by similar methods: namely, there is texture parallel to the growth direction with random grain orientation normal to this direction.

(2) The Alqloy (KCl-33 m/o KBr) was found to have low values of elastic moduli, a high value of Poisson's ratio, and a very minor amount of elastic anisotropy. Based on calculated values of moduli for pure polycrystalline KCl (approximately 2.4×10^{10} nt/m²) and the measured values of KCl-KBr alloys, one may conclude that additions of KBr to KCl result in appreciable decreases in the elastic moduli.

(3) Polycrystalline NaCl exhibits large deviations in the moduli from the calculated values. This result may be due to sample size.

(4) The experimental results obtained have shown that polycrystalline elastic properties calculated from single crystal data may yield reasonable estimates for these material parameters, but we cannot arbitrarily accept such values, particularly if significant texture is present.

It may be emphasized that the figure of merit criteria and ultimately the design criteria embody an inverse relationship between the Young's modulus and the fracture and/or yield stress. Consideration should be given in the design of processing procedures in order to produce a final window texture which would represent the best compromise between the strength and elastic parameters.

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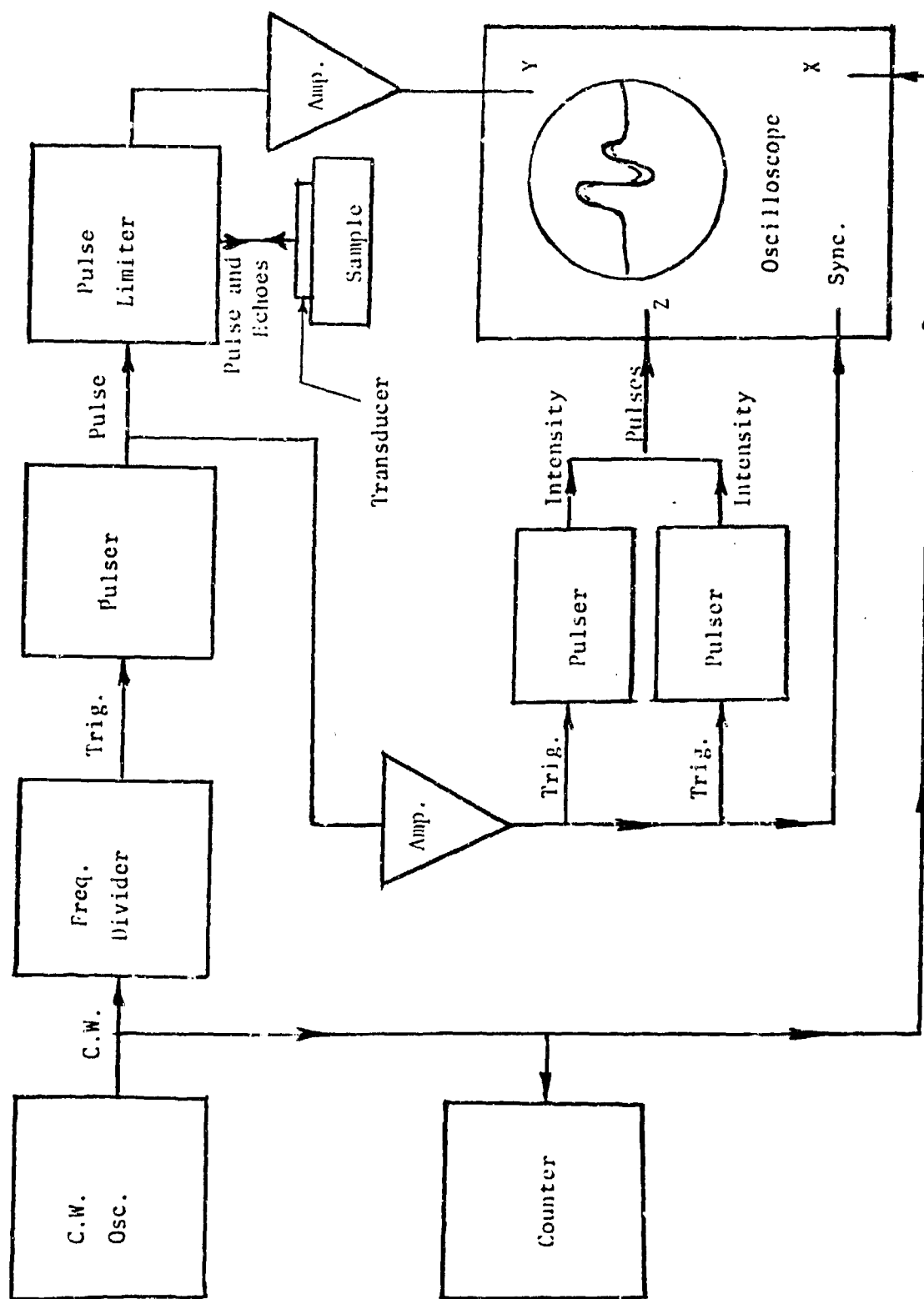
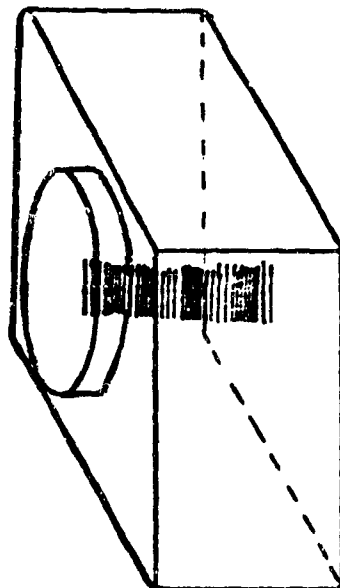
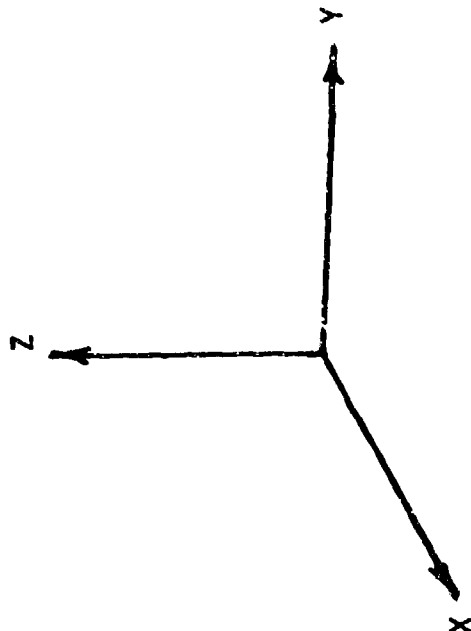
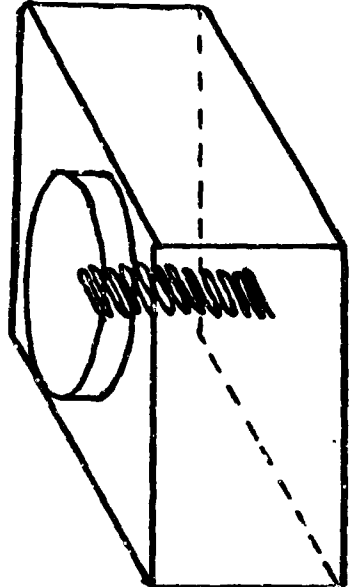
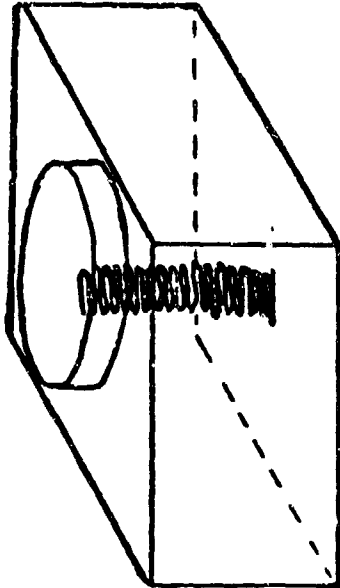


FIGURE 1. BLOCK DIAGRAM OF EQUIPMENT FOR DYNAMIC MODULUS MEASUREMENT.



(A) LONGITUDINAL



(B) SHEAR

FIGURE 2, ULTRASONIC WAVE MODES EMPLOYED FOR MEASUREMENT OF ELASTIC CONSTANTS.

TABLE 1

FIGURES OF MERIT FOR BULK ABSORPTION ONLY*

OPTICAL DISTORTION AT WAVELENGTH (λ)

$$\frac{\lambda}{\beta \chi} \cdot \frac{K}{R}$$

WATTS

$$\left(\frac{\lambda}{\beta \chi} \right) \cdot \left(\frac{K}{R} + \dots h_s \right)$$

WATTS

$$\left(\frac{\lambda}{\beta \chi} \right) \cdot \left(\frac{C R}{t} \right)$$

WATTS

$$\left(\frac{\sigma_c}{\alpha \beta E} \right) \cdot K$$

FRACTURE

EDGE-COOLED

$$\left(\frac{\sigma_c}{\alpha \beta E} \right) \cdot (K + \dots h_s R)$$

FACE-COOLED

$$\left(\frac{\sigma_c}{\alpha \beta E} \right) \cdot \left(\frac{C R^2}{t} \right)$$

PULSED

 χ IS AN OPTICAL DISTORTION PARAMETER

$$\chi = \frac{dn}{dt} + (1+\nu)\alpha(n-1) + \frac{1}{2}n^3\alpha E < \pi$$

* FRANK A. HERRIGAN, CONFERENCE ON HIGH POWER INFRARED LASER WINDOW MATERIALS, OCTOBER 1971.

TABLE 2

Measured Elastic Properties of CVD ZnSe^{*}

Direction of Wave Propagation	Direction of Shear Wave Polarization	Young's Mod. 10^{10} nt/m ²	Shear Mod. 10^{10} nt/m ²	Bulk Mod. 10^{10} nt/m ²	Poisson's Ratio
Z (growth dir.)	X	7.54	2.89	6.49	0.306
Z (growth dir.)	Y	7.54	2.88	6.49	0.307
X	Y	7.46	2.84	6.60	0.312
X	Z	7.61	2.91	6.500	0.305
Y	X	7.45	2.84	6.59	0.311
Y	Z	7.61	2.92	6.49	0.304

TABLE 3

Calculation of Polycrystalline Elastic Properties

of ZnSe from Single Crystal Data[†]

	Young's Mod. 10^{10} nt/m ²	Shear Mod. 10^{10} nt/m ²	Bulk Mod. 10^{10} nt/m ²	Poisson's Ratio
Voight	8.33	3.29	5.95	0.267
Hashin	7.76	3.02	5.95	0.283
Shtrikman	7.45	2.88	5.95	0.291
Reuss	6.80	2.60	5.95	0.310

^{*} Measured density = 5.265 gm/cc.

[†] Simmons and Wang (14).

TABLE 4

MEASURED ELASTIC PROPERTIES OF PIR ALLOY*

DIRECTION OF WAVE PROPAGATION	DIRECTION OF SHEAR WAVE POLARIZATION	YOUNG'S MOD. 10^{10} NT/M ²	SHEAR MOD. 10^{10} NT/M ²	BULK MOD. 10^{10} NT/M ²	POISSON'S RATIO
Z (DEFORM.DIR.)	X	1.60	.565	2.95	0.410
Z (DEFORM.DIR.)	Y	1.60	.566	3.00	0.411
X	Y	1.58	.559	2.95	0.411
X	Z	1.57	.554	2.96	0.412
Y	X	1.58	.556	2.92	0.410

* MEASURED DENSITY = 2.493 GM/CC.

TABLE 5

Measured Elastic Properties of PIR NaCl^{*}

Direction of Wave Propagation	Direction of Shear Wave Polarization	Young's Mod. 10^{10} nt/m ²	Shear Mod. 10^{10} nt/m ²	Bulk Mod. 10^{10} nt/m ²	Poisson's Ratio
Z (deform. dir.)	X	3.88	1.54	2.61	0.251
Z (deform. dir.)	Y	3.88	1.55	2.61	0.253
X	Z	3.51	1.36	2.77	0.290
X	Y	3.98	1.61	2.43	0.228
Y	Z	3.48	1.35	2.68	0.284
Y	X	4.04	1.68	2.24	0.200

TABLE 6

Calculation of Polycrystalline Elastic Properties
of NaCl from Single Crystal Data[†]

	Young's Mod. 10^{10} nt/m ²	Shear Mod. 10^{10} nt/m ²	Bulk Mod. 10^{10} nt/m ²	Poisson's Ratio
Voight	3.76	1.50	2.51	0.250
Hashin	3.72	1.48	2.51	0.253
Shtrikman	3.72	1.48	2.51	0.253
Reuss	3.67	1.46	2.51	0.256

^{*} Measured density = 2.168 gm/cc.

[†] Simmons and Wang (14).

TABLE 7

MEASURED ELASTIC PROPERTIES OF PIR KCL - 5 M/O KBr*

DIRECTION OF WAVE PROPAGATION	YOUNG'S MOD. 10^{10} NT/M ²	SHEAR MOD. 10^{10} NT/M ²	BULK MOD. 10^{10} NT/M ²	POISSON'S RATIO
Z (PRESSING DIR.)	1.92	0.689	3.04	0.395
X	1.85	0.662	2.92	0.395
Y	1.86	0.663	3.18	0.403

* MEASURED DENSITY = 2.028 GM/CC.

TABLE 8

SUMMARY OF ELASTIC PROPERTY DATA FOR
SELECTED WINDOW MATERIALS

<u>MATERIAL</u>	<u>YOUNG'S MODULUS</u>	<u>SHEAR MODULUS</u>	<u>BULK MODULUS</u>	<u>POISSON'S RATIO</u>
CVD ZnSe	7.54×10^{10} NT/M ²	2.88×10^{10} NT/M ²	6.49×10^{10} NT/M ²	0.307
	10.95×10^6 psi	4.18×10^6 psi	9.42×10^6 psi	
PIR ALQLOY	1.60	.566	2.98	0.411
	2.31	.820	4.32	
PIR NaCl	3.88	1.55	2.61	0.252
	5.62	2.25	3.79	
PIR KCl-5M/OKBr	1.92	.689	3.04	0.395
	2.79	.999	4.41	